

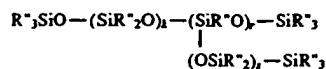
times observed even at the lowest (7.7 kg/hr) methanol flow rate. In most cases, there was no carryover of foam into the distillation columns because the reactor was designed with a long disengagement height.

In one experiment with a previously used batch of solvent which had been centrifuged at 1000 rpm for particulate separation, but not chemically treated for soluble silicate removal, foaming was visible when the methanol flow rate was increased from 7.7 kg/hr to 9.1 kg/hr. A solution containing 66 g FS 1265 in 594 g THERMINOL® 59 was then injected into the reactor and the response observed through the observation window. Defoaming was instantaneous and vapors were observed to rise from the surface of the slurry to the reactor outlet. No additional foaming occurred during the ensuing hours of reaction at methanol flow rates of 9.1–11.8 kg/hr. The quantity of FS 1265 used was equivalent to 322 parts per million based on the total weight of slurry. Untreated solvent from this experiment was recycled, without further purification, to the reaction of three additional 68 kg batches of silicon. Foaming was not observed in any of these runs. Subsequent experiments showed that FS 1265 concentrations as low as 50, 100 and 200 parts per million can provide effective and durable defoaming action in the 400 L reactor.

What is claimed is:

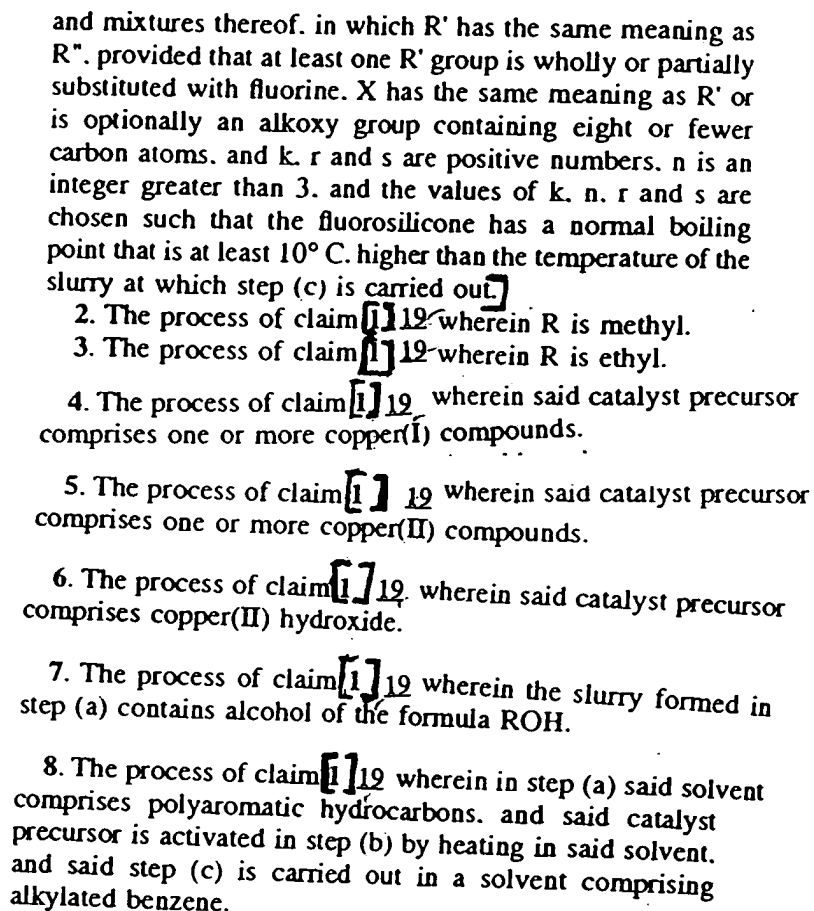
1. A process for producing trialkoxysilane of the formula  $\text{HSi(OR)}_3$ , wherein R is an alkyl group containing 1 to 6 carbon atoms inclusive, comprising

- (a) slurring silicon metal in a thermally stable solvent in the presence of a surface-active additive and of a catalyst precursor which is halogen-free and which comprises copper,
- (b) activating said catalyst precursor, thereby generating a catalyst for the reaction of step (c), and
- (c) reacting said silicon metal with an alcohol of the formula ROH in the presence of the catalyst generated in step (b) to form said trialkoxysilane; wherein said surface-active additive comprises (i) a mixture of (i)(A) hydrophobized silica particles with average particle sizes in the range of 0.2–5 microns, and specific surface area between 50 and 400 square meters per gram and (i)(B) an organopolysiloxane selected from the group consisting of compounds of the general formula:  $\text{R}''_3\text{SiO}-(\text{SiR}''_2\text{O})_n-\text{SiR}''_3$ , cyclic compounds of the general formula:  $(\text{R}''_2\text{SiO})_n$ , branched oligomers and polymers of the formula



and mixtures thereof, wherein R'' in each occurrence is the same or different and each is a  $\text{C}_1$ – $\text{C}_{20}$  alkyl group, phenyl, alkyl-substituted phenyl, cycloalkyl, or alkyl-substituted cycloalkyl; a, b, k, r and s are greater than zero and have values such that the normal boiling point of the organopolysiloxane is at least 10° C. higher than the temperature of the Direct Synthesis slurry; wherein the weight ratio of (i)(A) to (i)(B) is 1:99 to 99:1, or (ii) an organofluorosiloxane selected from the group consisting of compounds of the general formula  $\text{XR}'_2\text{SiO}-(\text{SiR}'_2\text{O})_n-\text{SiR}'_2\text{X}$ , cyclic compounds of the general formula  $(\text{R}'_2\text{SiO})_n$ , branched oligomers and polymers of the formula

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9. The process of claim 1<sup>19</sup> wherein step (b) comprises fully reducing to  $\text{Cu}^0$  copper in said catalyst precursor which is not fully reduced.

10. The process of claim 9 wherein step (b) is carried out by reacting said catalyst precursor with gaseous hydrogen.

11. The process of claim 9 wherein step (b) is carried out by reacting said catalyst precursor with carbon monoxide.

12. The process of claim 9 wherein step (b) is carried out by reacting said catalyst precursor with  $\text{SiH}_4$ .

13. The process of claim 9 wherein step (b) is carried out by reacting said catalyst precursor with an organosilane containing one or more  $\text{SiH}$  groups.

14. The process of claim 9 wherein step (b) is carried out by reacting said catalyst precursor with an organosilane containing one or more  $\text{SiH}_2$  groups.

15. The process of claim 9 wherein step (b) is carried out by reacting said catalyst precursor with an organosilane containing one or more  $\text{SiH}_3$  groups.

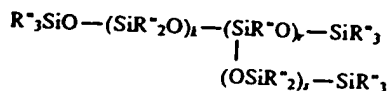
16. The process of claim 9 wherein the slurry formed in step (a) contains alcohol of the formula  $\text{ROH}$ .

17. The process of claim 9 wherein hydrogen is formed in the reaction of step (c) and all or a portion of said hydrogen is recycled to step (b) and is used in the reduction of step (b).

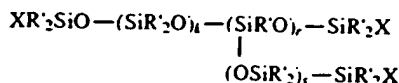
18. The process of claim 9 wherein in step (a) said solvent comprises polyaromatic hydrocarbons, and said catalyst precursor is reduced in step (b) by heating in said solvent, and said step (c) is carried out in a solvent comprising alkylated benzene.

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(i) an organopolysiloxane selected from the group consisting of compounds of the general formula  $R''_3SiO-(SiR''_2O)_n-SiR''_3$ , cyclic compounds of the general formula  $(R''_2SiO)_n$ , branched oligomers and polymers of the formula



(ii) an organofluorosiloxane selected from the group consisting of compounds of the general formula  $\text{XR}'_2\text{SiO}-(\text{SiR}'_2\text{O})_k-\text{SiR}'_2\text{X}$ , cyclic compounds of the general formula  $(\text{R}'_2\text{SiO})_n$ , branched oligomers and polymers of the formula



and mixtures thereof, wherein R' in each occurrence is the same or different and has the same meaning as R'' provided that at least one R' group is wholly or partially substituted with fluorine, X has the same meaning as R' or is optionally an alkoxy group containing eight or fewer carbon atoms, and k, r, and s are positive numbers, n is an integer greater than 3, and the values of k, n, r and s are chosen such that the organofluorosiloxane has a

normal boiling point at least 10°C higher than the temperature of the slurry during the reaction of step (c);  
or mixtures of (i) and (ii).

20. The process of claim 19, wherein the hydrophobized solid particles are silica.

21. The process of claim 19, wherein the catalyst precursor is halogen-free.

22. The process of claim 19, wherein hydrophobized solid particles have an average particle size in the range of 0.2-5 microns.

23. The process of claim 19, wherein hydrophobized solid particles have a specific surface area between 50 and 400 square meters per gram.

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